TEST PROCEDURE

TP 118	
Title Analysis of Benzene in Gasoline by Gas Chromatography	**Page Number** 1 of 20
Originator Thomas Woodside	**Supersedes** none
Responsible Organization Fuels and Chemical Analysis Branch	**Computer Program** HP Chemstation Software
Type of Test Report Computer-generated report	**Data Form Number** none
Report Distribution Test Requestor	**Implementation Date** 07-10-95

Implementation Approval

Test Procedure Authorized by EPCN #181

Revision Description

Note: Specific brand names in EPA/EOD procedures are for reference only and are not an endorsement of those products.

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Attachments

1. Purpose

This procedure measures benzene in gasoline samples that may contain ethanol and/or other oxygenated compounds. The method is applicable to measuring benzene in gasoline samples in the range of 0.01 to 5.00 % volume/volume (v/v). Samples containing more than this amount may be diluted to fall within the range.

2. Test Article Description

Gasoline samples, containing between 0.01 % (v/v) and 5.00 % (v/v) benzene and up to 12% (v/v) ethanol. Oxygenates other than ethanol have not been found to interfere with this benzene analysis and are not restricted in quantity.

3. References

3.1 "Code of Federal Regulations," Title 40, Part 80, Section 46

3.2 Quality Assurance Management Staff, "Calculation of Precision, Bias, and Method Detection Limit for Chemical and Physical Measurements," U.S. EPA, March 30, 1984

3.3 American Society for Testing and Materials (ASTM) D 3606-92: "Standard Test Method for Determination of Benzene and Toluene in Finished Motor and Aviation Gasoline by Gas Chromatography," and modifications as presented in Question and Answer document dated November 1994

3.4 "Hewlett-Packard 5890A Instrument Manual"

3.5 "Hewlett-Packard Chemstation Manual"

3.6 ASTM Manual MNL7, "Manual on the Presentation of Data and Control Chart Analysis: 6th Edition"

3.7 Wasson – "ECE Instrumentation Manual"

3.8 Environmental Protection Agency (EPA) Engineering Operation Division (EOD) "TP109, Chain of Custody Procedure"

4. Required Equipment

4.1 2-milliliter (mL) sample vials or vials suitable for the autosampler with aluminum crimp caps and lined rubber septa

4.2 40-mL vials or vials for calibration standards and samples

4.3 Class A glass pipets and volumetric flasks (various sizes) for preparation of calibration standards, check standards, analytical samples, and control fluid samples

4.4 Gas chromatograph (GC) equipped with a backflush valve and a thermal-conductivity detector (TCD)

Equipment Used: Hewlett-Packard 5890-A equipped with the following columns, in order:

5' X $^1/_8$" Methyl Silicone on Chromasorb (10% OV101 on Chrom PAW 80/100)

5' X $^1/_8$" TCEP on Chromasorb (20% TCEP on Chrom PAW 80/100)

15' X $^1/_8$" Carbowax 1540 (15%) on Chromasorb W (e.g. Wasson-ECE K16)

4.5 Computer system for controlling the analysis and collecting data

Equipment Used: Hewlett-Packard Vectra 486/66XM with Hewlett-Packard Chemstation software

4.6 GC injection syringe, 5-microliter (μL) volume, or autosampler

4.7 Reagent grade ethanol, benzene, and sec-butanol

4.8 Benzene-free iso-octane to prepare standards for calibration

4.9 Helium; ultra pure grade (used as GC carrier gas)

4.10 Compressed nitrogen for valve operation

4.11 “Chromatograph Calibration Curve Report”

4.12 Form 109-01, “Control Chart for the Range of Duplicate Analyses”

4.13 Form 109-02, “Control Chart for Individual Observations”

4.14 Form 3500-5, “Fuels Field Inspection”

4.15 Independent Standard:

National Institute of Standards and Technology (NIST) standards are not available for this analysis, so an independent standard is prepared by a different analyst using a different lot number of pure benzene from that used to produce the calibration standards. Production is otherwise identical to production of calibration standards.

4.16 Control fluid:

California Phase II test fuel drawn from the National Vehicle and Fuel Emissions Laboratory (NVFEL) underground storage tanks is used as a control fluid. The concentration of benzene in each batch of this fluid must be measured against calibration quality standards before use.

4.17 Calibration standard:

Calibration standards are made from pure compounds diluted in benzene-free blank stock to the desired concentration. They are used for the purpose of calibrating the instrument.

4.18 Check standard:

Check standards are prepared in exactly the same manner as calibration standards, and may be identical to the calibration standards except that they are used to check the calibration after it has been established.

4.19 Internal standard:

The internal standard is sec-butanol. It is added to each sample to be analyzed to allow for compensation of sample injection quantity variation and other sampling factors during analysis.

4.20 Blank stock:

The blank stock is a mixture of 10% (v/v) benzene-free ethanol in benzene-free iso-octane. It is used as a diluent in standards and for reagent blanks.

4.21 Reagent blank:

A reagent blank is a sample prepared by the analyst that contains only blank stock and internal standard. It is used to verify that the blank stock and internal standard contain no benzene and that benzene is not introduced through the normal preparation of standards.

5. Precautions

5.1. Gasoline, benzene, and oxygenated compounds are extremely flammable and may be toxic when exposure is over a prolonged period. Benzene is a carcinogen. Persons performing this procedure must be familiar with the chemicals and hazards involved.

5.1.1 Dilutions must be performed in well-ventilated areas, preferably in a fume hood, away from open flames and sparks.

5.1.2 For fires or spills, specific instructions are available from the supervisor and/or Safety Officer.

5.1.3 Prior to performing the analysis, the analyst must be familiar with the Material Safety Data Sheets (MSDS) for materials used in this analysis.

5.2 Gas cylinders must be properly secured and handled with extreme caution because of the high volumes contained and the high pressures at which they are contained.

5.3 Because of the dependence of the density of reference materials on temperature, the preparation of standards and samples must be done with all materials at lab temperature using Class A glassware.

6. Visual Inspection

6.1 Ensure that the sample vials are sealed and that the seals are intact. Make sure that they are properly labeled with the test number and other identification.

6.2 Visually inspect the GC and the autosampler injection syringe for correct setup.

6.2.1 Ensure that rinse and waste vials are installed and filled, if necessary.

6.2.2 Ensure that required reagents and solvents are available for use.

6.2.3 Ensure that the syringe and injection needle are installed and in working condition.

6.2.4 Ensure that there are adequate quantities of GC supply gases to maintain the run. At least 200 pounds per square inch (psi) in all supply cylinders is prudent.

7. Test Preparation

7.1 For packed column operation of the GC, set the flows to the manufacturer's specifications to achieve the separations desired. The following are guidelines for setting these parameters.

7.1.1 Set the injector pressure to 86 psi.

7.1.2 Set "Valve #1" to position "two" (on).

7.1.3 With the reference gas to the TCD turned off, set the gas flow rate at the exhaust of the TCD to 35 mL/minute using variable restrictor 2 (VR2).

7.1.4 Turn on the reference gas to the TCD.

7.1.5 Using the set screw inside the "TCD REF GAS" knob, set the total flow through the TCD to 75 mL/min.

7.1.6 Move the flow meter to the vent connected to variable restrictor #3 (VR3). Adjust VR3 to obtain a flow of 35 mL/minute.

7.1.7 Set the "injector A" temperature to 250 °C.

7.1.8 Set the "auxiliary temperature" to 150 °C to heat the valve and columns.

7.1.9 Set the detector temperature to 200 °C.

7.1.10 Turn on the TCD detector ("DET A").

7.1.11 Check the instrument clock, date, and time and correct if necessary.

7.2 Upon initial setup, or when it is determined that peak retention times have drifted due to column age or other causes, determine if the backflush valve timing remains between the i-octane and the n-nonane peaks.

7.2.1 Prepare a mixture of 5% (v/v) iso-octane in n-nonane.

7.2.2 Ensure that the injection valve is in the forward flow mode and inject 1 µL of this mixture into the injection port .

7.2.3 View the chromatagram using the HP Chemstation software. Note the time from the injection to the completion of the iso-octane peak and from the injection to the beginning of the n-nonane peak. Subtract the iso-octane time from the n-nonane time. The center of this range is the backflush valve activation time for the analysis. Enter this time in the appropriate section of the "Valve Timing Screen."

7.3 Determine if an instrument calibration is required. The following are guidelines for this determination:

If it is a new instrument or one not previously used for this analysis.

If the current calibrations are lost or damaged.

If it is after reconfiguration or repair of an existing instrument.

If a sample concentration is out of the range of current calibrations.

If the current check standard or control fluid results fall outside the acceptance criteria.

If calibration is not required, proceed to Section 8 of this procedure.

If calibration is required, complete the remaining steps in Section 7.

7.4 Prepare the calibration standards. Calibration standards are created by appropriate dilution of a high-concentration standard. The high-concentration standard is made by diluting pure compounds in benzene-free blank stock. Dilutions must be done with all materials at lab temperature using Class A glassware.

The following represents one dilution scheme for preparing a set of calibration standards. Other dilution schemes may be used, provided they are consistent with the range and concentration coverage presented here. Typical gasoline enforcement analysis covers the range 0.05% (v/v) to 5.0% (v/v) benzene with seven standards.

7.4.1 The high-concentration standard is made by volumetrically diluting 10 mL benzene, 20 mL ethanol, and 40 mL toluene to 200 mL with iso-octane. Toluene and ethanol are added volumetrically to provide reference peaks.

7.4.2 Subsequent standards are prepared by dilution of this mixture with calibration blank stock. The following dilution scheme is typically employed:

Volume of Intermediate (mL)	Flask Volume (mL)	Benzene Concentration (% V/V)	Toluene Concentration (% V/V)
50	100	2.50	10.0
25	100	1.25	5.0
15	100	0.75	3.0
7	100	0.35	1.4
3	100	0.15	0.6
1	100	0.05	0.2

7.5 Prepare the calibration standards for GC calibration.

7.5.1 Pipet 25 mL of standard into a 40-mL vial and add 1 mL of sec-butanol internal standard.

7.5.2 Mix each standard and fill an autosampler vial with it using a disposable pipet.

7.5.3 Label each vial with the benzene concentration and load it into the autosampler.

7.6 Check the instrument setup for the calibration run.

7.6.1 The calibration fit should be selected as linear, forced through the origin.

7.6.2 Verify that the retention time window has been set properly. The typical value is ±5% (this is the Chemstation default).

7.6.3 Check recent analyses for ethanol, benzene, sec-butanol, and toluene retention times and edit the correct value for each into the Chemstation calibration table if necessary. Refer to the Chemstation instruction manual for details of this operation.

7.6.4 The instrument should be calibrated with a range of standards that will encompass the likely concentration of the samples.

7.7 Initiate the calibration analysis according to the HP Chemstation Manual.

7.8 Following the completion of a calibration analysis, analyze the data using the "Chromatograph Calibration Curve Evaluation Report" report to verify the viability of the calibration curve. Any of the composite points that differ from the linear calibration curve by the greater of 0.06% absolute or 3% of point should be investigated. Analysis cannot proceed until the cause is resolved.

7.9 If the linearity of the calibration is satisfactory, add internal standard to the independent standard and analyze it.

If the difference between the result of this analysis and the named concentration of the standard exceeds the larger of 5.0% of the named concentration of the standard or 0.05% absolute, investigation, correction, and successful reanalysis are required before proceeding.

7.10 If the analysis of the independent standard is satisfactory, add 1 mL of internal standard to the control fluid and analyze it. The result of this analysis should be within the control chart limits.

If it is not within these limits, the calibration is considered suspect and the cause of the discrepancy must be resolved before the calibration can be accepted or samples analyzed.

7.11 If the data from this analysis appear satisfactory, the calibration must be saved within the data acquisition system.

7.11.1 The naming convention for saving a calibration is a prefix BNZ plus a date, written as month and day. For example, if a calibration were completed on the twenty-ninth day of December, it would be saved as "BNZ12-29."

7.11.2 After saving the calibration, a plot of the calibration should be printed, along with a list of the calibration parameters. This printout must be saved, along with the results of the independent standard analysis and the "Chromatograph Calibration Curve Evaluation Report" report, in the laboratory notebook specific to the instrument. This notebook is stored in a secure place, normally in close proximity to the instrument.

7.11.3 Note the fact of a new calibration on the control fluid control chart.

8. Test Procedure

101 Pipet 25 mL of each sample into individual 40-mL disposable vials.

102 Pipet 1 mL of sec-butanol internal standard into each container.

103 Seal each container and mix the contents. Mark each container with the sample number.

104 Using a disposable pipet, fill each autosampler vial with sample. Close each vial according to the manufacturer's instructions and label it with the sample number.

105 Create a sequence file for the Chemstation per the Chemstation instruction manual. Arrange the standards and test samples in the autosampler as described in the table below, and then input the sample and standard IDs into the data system.

Control fluid
Check standard
Reagent blank
Control fluid
Samples (10)
Duplicate sample
Control Fluid
Samples (10)
Duplicate sample
Control Fluid
Etc.

A minimum of one check standard and one reagent blank are used for each analysis session. The analytical session always begins and ends with the control fluid sample.

106 Operate the autosampler per the manufacturer's instructions to initiate the analysis.

107 At the completion of each analysis, the HP Chemstation will produce an output report showing the chromatogram and calculated concentrations. Review the individual GC reports to verify that all retention times are within the retention time window by verifying that each peak of interest is identified.

108 Ensure that the difference between the reported concentration of the check standard and its nominal concentration is less than the larger of 0.06% absolute or 3% of point.

If it is not within this limit, discard the vial and replace it with a fresh one.

If a fresh vial is out of specification, investigate the cause; it may be necessary to make new standards and recalibrate. See section 7.4

109 On Form 109-02, record and plot the result of the control fluid analysis. Confirm that the benzene concentration of the control fluid is within the appropriate control chart limits. Investigate any out-of-control conditions and take immediate corrective action. Record the action and its implications on the control chart and repeat this step.

If out-of-control occurrences cannot be reconciled, consult the laboratory supervisor.

If the investigation indicates that an out-of-control occurrence may have affected previously analyzed samples, repeat the samples analyzed since the last acceptable control fluid analysis. Discard the original results from these samples.

Initial the control chart in close proximity to the current entry, and save the chart in the instrument notebook.

Note: Subsequent charts should be placed behind the prior chart (if this is not first) and following the current calibration printout. The instrument notebook is stored in a secure location near the instrument.

110 On Form 109-01, record and plot the absolute difference (range) between duplicate analyses. Verify that the range of the duplicates is within the control chart limits. For this procedure, these chart limits are defined as the 3-sigma confidence limit for the range of duplicates, based on acquired data.

If the duplicate range is out of control, halt the analysis and take immediate corrective action to resolve the abnormal analysis variability. Record the action and its implications on the control chart.

If out-of-control occurrences cannot be reconciled, consult the laboratory supervisor.

When the problem is resolved, repeat the duplicate analysis and record and plot the results of this analysis on Form 109-01. Ensure that the results are within the control limits before proceeding with the analysis.

If the investigation indicates that an out-of-control occurrence may have affected previously analyzed samples, repeat the samples analyzed since the last acceptable duplicate analysis. Discard the original results from these samples.

Initial the control chart in close proximity to the current entry, and save the chart in the instrument notebook.

111 If an unknown sample is found to be above 5.0% (v/v) benzene, it must be volumetrically diluted to fall within the specified range of 1.0% to 5.0% (v/v) benzene. Samples found to contain ethanol should be diluted with blank stock. Samples containing no ethanol should be diluted with iso-octane. The diluent in either case must be analyzed prior to dilution to verify that it contains no detectable quantity of benzene. All dilutions must be done with Class-A volumetric glassware.

If the dilute analysis is satisfactory, calculate the concentration of benzene in the undiluted sample as follows:

$$B = B_b * [(V_S + V_O) / V_S]$$

where: B_b = concentration of benzene in the diluted sample

V_S = Volume of the original sample

V_O = Volume of the diluent

This calculation must be shown on the instrument-generated sample analysis report for the diluted sample.

112 If all acceptance criteria are met, the analyst transfers the sample results to Form 3500-5.

113 Discard the contents of the sample vials into an approved disposal container.

9. Data Input

9.1 The identity of the sample vials and the internal standard volume are input to the data system before the start of each chromatograph run.

9.2 If all acceptance criteria are met, the operator signs the analysis report.

9.3 The analyst transfers sample results to Form 3500-5.

9.4 Control fluid results are recorded and plotted on Form 109-02.

9.5 The results of duplicate analyses are recorded on Form 109-01.

10. Data Analysis

10.1 The instrument calculates the concentration of each sample based on the active calibration curve and the benzene response with reference to the internal standard response.

10.2 Upon completion of each discrete analysis, a summary report is generated by the GC data system (see Attachment B). The operator must verify that the identity and label on the chromatogram match the sample identification number from the vial.

10.3 The analyst reviews the individual reports to verify peak recognition, baseline stability, accuracy of the standard and control fluid measurements, and peak morphology. For this analysis, peaks should be reasonably symmetrical with no obvious splitting, flat tops, or baseline aberrations. Excessive trailing is an indication of column decay or other instrument problems.

10.4 Analysis results for diluted samples are corrected on the report form, per the formula in Step 111, to reflect their undiluted concentration by multiplying by the dilution factor.

10.5 The analyst reviews the results of the analyses of samples, control fluids, check standard, duplicate samples, and independent standard for conformance to acceptance criteria.

10.6 Following the completion of a calibration analysis, analyze the data using the "Chromatograph Calibration Curve Evaluation Report" report to verify viability of the calibration curve. Any of the composite points that differ from the linear calibration curve by 0.06% absolute or more than 3% of point should be investigated, and analysis cannot proceed until the cause is resolved.

11. Data Output

11.1 If a new calibration curve is generated, a plot of the calibration, a calibration parameter printout, and the "Chromatograph Calibration Curve Evaluation Report" report are filed, along with the results of the independent standard analysis, in a notebook specific to the instrument.

11.2 Individual sample analysis chromatograms and reports containing concentrations of discrete analyses are output from the HP Chemstation.

If this is the last analysis type scheduled, the result outputs and FFV go to the data entry box, if not, the forms follow the samples to the next scheduled analysis.

11.3 Results of control fluid and duplicate analyses are recorded on the appropriate control charts. These are saved in a notebook specific to the instrument. This notebook is stored in a secure place, normally in close proximity to the instrument. Original outputs of control fluid analyses are discarded.

12. Acceptance Criteria

12.1 The ethanol, benzene, sec-butanol, and toluene GC retention times for the check standard and control fluid analyses must be within the recognition time window of the current calibration, as evidenced by instrument peak identification.

12.2 The composite points of the calibration curve must not differ from the curve by more than 0.06% absolute or 3.0% of point.

12.3 If a new calibration curve is generated, then the difference between the result of the analysis of the independent standard and the named concentration of that standard must not exceed the larger of 5.0% of the named concentration of that standard or 0.05% absolute.

12.4 The difference between the reported concentration of the check standard and its nominal concentration must be less than the larger of 0.06% absolute or 3% of point.

12.5 Concentrations of the control fluid samples must be within the limits established by the control charts.

12.6 Measured concentration differences for the duplicate samples must be within the limits established by the control charts. Each chart is initialed by the analyst in close proximity to the appropriate point.

Failure to meet this criterion requires investigation and corrective action. Samples analyzed since the last successful duplicate analysis must be reanalyzed.

12.7 The baseline of any analysis run must not contain any noise greater than the threshold level for the lowest calibrated point and have no discernible drift. Individual peaks should not display any excessive tailing or other aberrant morphology.

12.8 The “Fuels Field Inspection” forms must be initialed by the analyst to signify they have been checked for compliance with the acceptance criteria.

12.9 If a new calibration curve is generated, the control fluid sample analyzed immediately after the calibration must also conform to the control chart limits in Step 7.10.

12.10 The blank stock analyzed as part of the calibration and used for dilutions must have no detectable amount of benzene.

13. Quality Provisions

13.1 Visual inspection of samples and instrument.

13.2 Inspection of each GC sample trace for peak identification and resolved peaks.

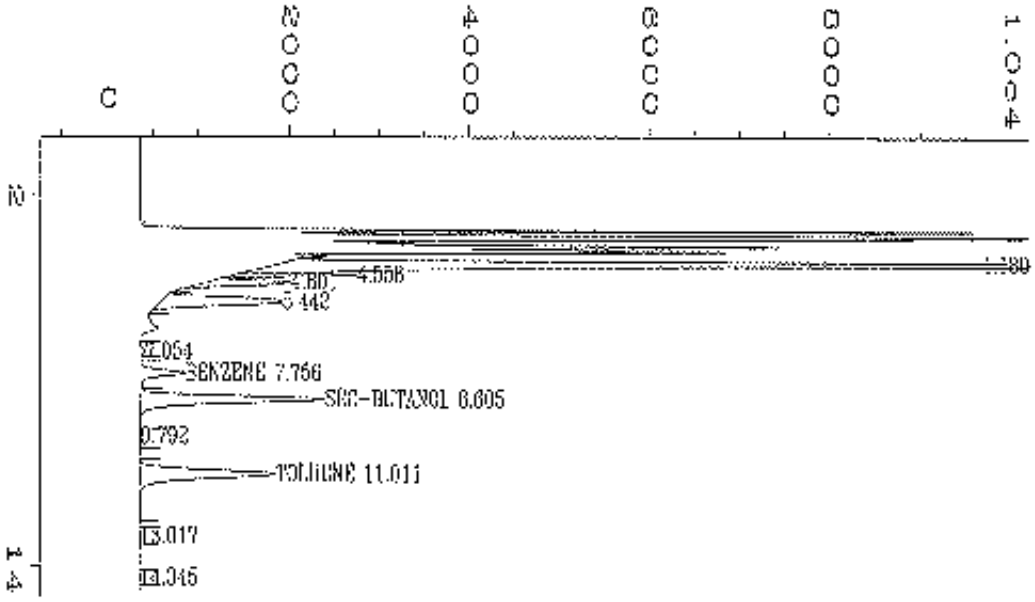
13.3 Check standard analysis before test sequence to assess instrument calibration.

13.4 Analysis of control fluid to assure calibration accuracy and the accuracy of internal standard addition.

13.6 Analysis of reagent blanks to assess reagent purity.

13.7 Analysis of “independent” standards to verify the accuracy of the calibration.

Gas Chromatograph Internal Standard Report



Internal Standard Report

Data File Name : C:\HPCHEM\1\DATA\1mar95L\001F0101.D
Operator :
Instrument : INSTRUMEN
Sample Name : xxx
Run Time Bar Code:
Acquired on : 30 Jan 95 02:00 PM
Report Created on: 30 Jan 95 02:17 PM
Last Recalib on : 20 NOV 94 10:04 AM
Multiplier : 1

Page Number : 1
Vial Number : 1
Injection Number : 1
Sequence Line : 1
Instrument Method: 3606.MTH
Analysis Method : 3606.MTH
Sample Amount : 0
ISTD Amount : 3.85

Sig. 1 in C:\HPCHEM\1\DATA\1mar95L\001F0101.D

Ret Time	Area	Type	Width	Ref#	ml	Name
6.584	* not found *			1		ETHANOL
7.756	6647	BV	0.216	1	0.755	BENZENE
8.605	32435	VV	0.247	1-I	3.850	SEC-BUTANOL
11.011	29213	BB	0.300	1	3.463	TOLUENE

Method: test\seq

Sequence Recalibration Table

Cal. Line	Cal. Level	Update Response Factor	Update Retention Times	Recalib Interval

Signal Plot Information

Signal	Attn. (2^)	Offset (%)	Time (Min.)
1	0	10	10
2	4	10	10

Integration Events

Events:	Value:	Time:
Initial Area Reject	1	INITIAL
Initial Peak Width	0.040	INITIAL
Shoulder Detection	OFF	INITIAL
Initial Threshold	-12	INITIAL
Integrator OFF		0.000
Integrator ON		4.000

Report Specification

Destination:	Report/Chromatogram to Printer
Based on:	Area
Calculations:	ISTD
Printer Output:	Combined Chromatogram and Report
Report Header:	On Each Report Page

Graphics Options

Title:	Vertical
Include:	
Axes Units:	On
Peak Names:	On
Retention Times:	On
Baselines:	On
Tick Marks:	On
Peak Labels Font:	Default 12

Calibration Table

Pk#	RT	Lvl	ml	Amt/Area	Ref Istd	I#	Name
1	6.584	1	10.0	1.1093e-004		1	ETHANOL
		2	5.0	1.1281e-004			
		3	2.5	1.1642e-004			

Method: C:\HPCHEM\1\METHODS\3606.MTH

		4	1.25	1.2281e-004		
		5	0.63	1.3452e-004		
		6	0.31	1.2923e-004		
		7	0.15	1.3583e-004		
2	7.757	1	5.0	1.1153e-004		1 BENZENE
		2	2.5	1.138e-004		
		3	1.25	1.1644e-004		
		4	0.63	1.2055e-004		
		5	0.31	1.219e-004		
		6	0.15	1.1269e-004		
		7	0.07	1.1296e-004		
3	8.605	1	3.85	1.2048e-004	ISTD	1 SEC-BUTANOL
		2	3.85	1.211e-004		
		3	3.85	1.2066e-004		
		4	3.85	1.2053e-004		
		5	3.85	1.2107e-004		
		6	3.85	1.1938e-004		
		7	3.85	1.194e-004		
4	11.018	1	20.0	1.2232e-004		1 TOLUENE
		2	10.0	1.1977e-004		
		3	5.0	1.1961e-004		
		4	2.5	1.2044e-004		
		5	1.25	1.219e-004		
		6	0.63	1.2116e-004		
		7	0.31	1.1849e-004		

Calibration Settings

Title:

Reference window:	5.000 %
Non-reference window:	5.000 %
Units of amount:	ml
Multiplier:	1.0
RF uncal peaks:	0.0
ISTD# to adjust uncal peaks:	0
Sample Amount:	0.0

Sample ISTD Information

I#	Amount
1	3.85

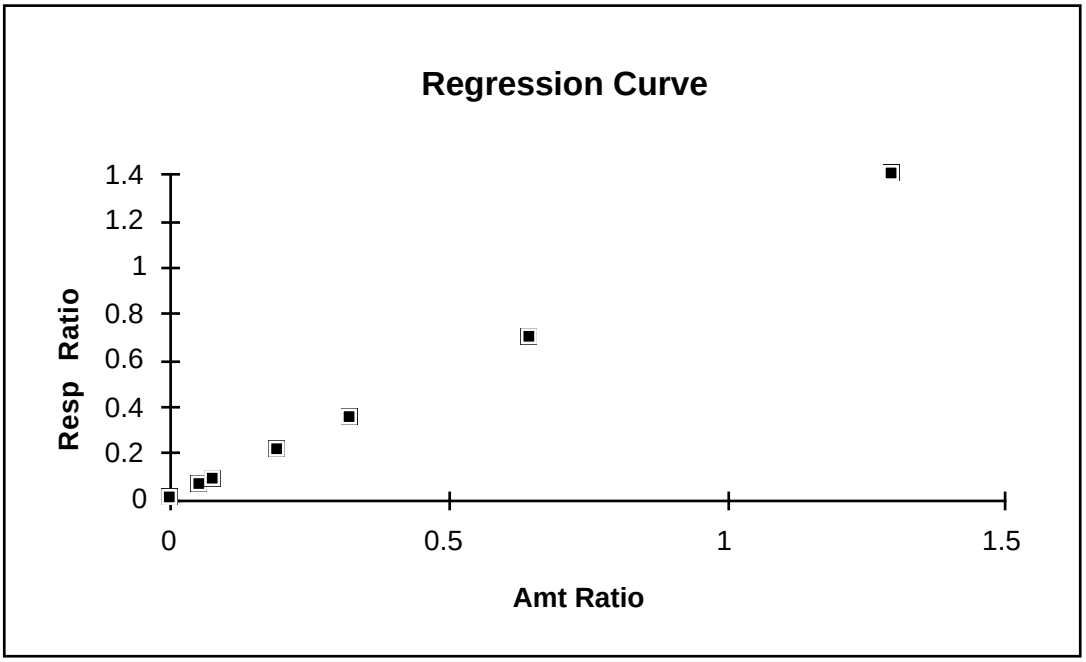
Multilevel Information

Fit: Linear
Origin: Force

Chromatograph Calibration Curve Evaluation Report

	INPUT DATA						EVALUATION DATA		
	Calib. Table		Avg from Cal Runs		Curve Coefficients * {rsp rat=M(amt rat)+B}				
Calib. Level	Nom. Conc. (%)	ISTD Conc. (%)	Analyte Area (Counts)	ISTD Area (Counts)	Slope (M)	Intercept (B)	Curve Calc Conc (%)	Δ (Calc-Nom)	%Δ (Δ/Nom)
1	0.05	3.85	394	32906	1.07	0.0115	0.00	-0.05	-96.6%
2	0.15	3.85	2172	32954			0.20	0.05	30.5%
3	0.35	3.85	3035	32497			0.29	-0.06	-15.8%
4	0.75	3.85	7038	32779			0.73	-0.02	-2.5%
5	1.25	3.85	11642	32748			1.24	-0.01	-1.0%
6	2.50	3.85	22844	32611			2.48	-0.02	-0.8%
7	5.00	3.85	46127	32959			4.99	-0.01	-0.1%

* To evaluate calibration curves using the HP Chemstation "FORCE" option, coefficients from a dummy curve generated with the same points using the HP Chemstation "IGNORE" option must be used.



Procedure Number _____

Date _____

Analyst _____